Effects of biosolids application on soil chemical properties in peri-urban agricultural systems

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Abstract. The application of biosolids as a fertilizer in agricultural cultivation are common practices in many countries. This study investigates the effects of such practices in field experiments on Fluvisol and Acrisol soils in peri-urban Hanoi City. We compared biosolid-fertilized vegetable/rice-dominated systems (biosolids applied as chicken manure (Acrisol in Vinh Phuc Province) or composted pig manure mixed with rice straw (Fluvisol in Ha Tay Province). The biosolids were applied at six different rates representing from 0 to 450% of the normal annual dose used by local farmers. The application of biosolids had highly significant positive effects on organic carbon (TOC%) and total nitrogen (N_{tot}%), when the six different treatments of composted manure and chicken manure were compared. The soil reverse *aqua regia*-extractable (Rev *Aq Reg*) Zn increased linearly with biosolids application rate at both sites and the linear regression showed Zn (mg kg^{-1}) = 112.5 + 13.25×10^{-3}×composted manure (t ha^{-1}) (r^2=0.58) or Zn (mg kg^{-1}) = 43.77 + 35.04×10^{-3}×chicken manure (t ha^{-1}) (r^2=0.73), whereas Cu (Rev *Aq Reg*) only increased significantly at the Vinh Phuc site. The Cd and Pb (Rev *Aq Reg*) concentration is not clearly different from the control after short-term (one time) application of biosolids. The application of biosolids increased the EDTA-extractable fraction of Cd, Cu and Zn, but had no effect on NH_4NO_3-extractable fractions of these elements.

Keywords: Chicken manure; Composted manure; Biosolids; Heavy metals; Trace elements.

1. Introduction

There is a growing concern about the risk of contamination of waters, soils and agricultural products, in the rapid urbanizing areas in the South-East Asia due to the heavy, or inappropriate, use of organic wastes, fertilizers, pesticides, and poor quality irrigation water [13]. Urbanization and industrialization processes always lead to increased production of waste, i.e. wastewater and solid waste. Industrial, agricultural and domestic effluents, such as biosolids and wastewater, are either

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dumped on land or used for irrigation and fertilization purposes, what creates both opportunities and problems [33].

The advantages of reusing waste are that it provides a convenient disposal of waste products and has the beneficial aspects of adding valuable plant nutrients and organic matters to soil [10]. Biosolids is a beneficial soil amendment, especially for arable soils of inherently low organic matter content, as it may improve many soil properties, such as pH and the contents of organic matter and nutrients [10, 23, 29]. However, as wastes are products of human society, generally enhanced concentrations of potential toxic substances including trace metals, which may limit the long-term use of effluents for agricultural purposes due to the likelihood of phytotoxicity, health and environmental effects [17, 33]. Even after a short-term application of biosolids, the level of trace metals in soils can increase considerably [21, 24].

If the content of trace metals increases above a certain critical concentration due to their accumulation in soil, it can have negative environmental effects, which can include negative effects on soil biota and hence on microbial and faunal activity [7]. Furthermore, trace metals can affect crop growth and quality, and thus pose risks for human health [4, 16]. Therefore, the risk of contamination by trace metals must be considered when biosolids are applied and the understanding of the behaviour of metals in the soil is essential for assessing environmental risks when the wastes are applied in agro-ecosystems.

The main objective of this paper was to quantify the effects of reuse of biosolids (in the form of animal manure) as nutrient sources by: (i) investigating the effects of biosolids application on soil pH, EC, organic carbon, total nitrogen and trace metals (cadmium (Cd), copper (Cu), lead (Pb) and zinc (Zn)); (ii) investigating the effects of application of biosolids especially as regards trace metal accumulation and solubility.

2. Materials and methods

2.1. Location of the research areas

Soil samples were collected from peri-urban areas of Hanoi City including Ha Tay and Vinh Phuc provinces (Table 1). The sampled areas are located in delta and lowland areas with a tropical monsoon climate. The annual rainfall is 1500-2000 mm, more than 50% of which are concentrated during the period from June to August. The mean monthly temperature varies between 17 and 29^{o}C, with the warmest period from June to August and the coldest during December and January.

2.2. Biosolids application

Organic fertilizers (biosolids) have been used in agriculture in Vietnam for a long time. In this study, field experiments with different rates of biosolids application to agricultural soil were set up in a collaboration between the National Institute for Soils and Fertilizers (NISF) and CSIRO Land and Water Australia within an ACIAR project in Ha Tay and Vinh Phuc provinces [26]. Biosolids in the form of chicken or pig manures co-composted with rice straw (composted manure) were applied at six different levels ranging from 0% to 450% of what local farmers normally apply per year: 20 tones ha^{-1} (100%) for chicken manure and 14 tones ha^{-1} (100%) for composted manure (Table 1). The experiments had a randomized block design with triplicates of the treatments. The biosolids were characterized prior to the application (Table 2). The total organic carbon content in chicken manure was higher than that in composted manure. Cadmium, Cu and Zn concentrations in chicken manure and composted manure were higher than ‘total’ (reverse *aqua regia*) concentrations of these metals in the experimental soils [8].

In Ha Tay Province, the soil type is a Cambic Fluvisol [5] and the composted manure was applied in February 2003. The crop here was rice (*Oryza sativa* L.). At the Vinh Phuc site, where the soil type is a Haplic Acrisol [5], the chicken manure was applied in November 2002. Vegetables were cultivated in the experiment, mainly cabbage (*Brassica oleacea* L.) and squash (*Benicasa hispida* L.). At both sites, soil sampling was carried out in June 2004.

Table 1. Description of biosolids treatments (randomized block design with three replicates) in experiments on Fluvisols and Acrisols in the peri-urban areas of Hanoi City

No.	Location / soil types	Treatments	Geographic coordinates	Name	Crop	Time of experiment
1	Ha Tay / Fluvisols	Composted manure[a]	N: 21°6.02' E: 105°40.78'			
		0 t ha^{-1}		BoDp1	Rice	17 months
		7 t ha^{-1}		BoDp2	Rice	17 months
		14 t ha^{-1}		BoDp3	Rice	17 months
		21 t ha^{-1}		BoDp4	Rice	17 months
		42 t ha^{-1}		BoDp5	Rice	17 months
		63 t ha^{-1}		BoDp6	Rice	17 months
2	Vinh Phuc / Acrisols	Chicken manure[a]	N: 21°9.02' E: 105°45.07'			
		0 t ha^{-1}		BoMl1	Cab/squ[b]	20 months
		10 t ha^{-1}		BoMl2	Cab/squ	20 months
		20 t ha^{-1}		BoMl3	Cab/squ	20 months
		30 t ha^{-1}		BoMl4	Cab/squ	20 months
		60 t ha^{-1}		BoMl5	Cab/squ	20 months
		90 t ha^{-1}		BoMl6	Cab/squ	20 months

[a] Fresh weight. Farmers usually apply biosolids at the rate of 14 t ha^{-1} yr^{-1} for composted manure and 20 t ha^{-1} yr^{-1} for chicken manure. Experimental design included only one application at the beginning of the experiment.
[b] cab/squ: Cabbage (*Brassica oleacea* L.) and squash (*Benicasa hispida* L.)

2.3. Soil sampling strategy and sample preparation

For assessment of the impact of biosolids on agricultural soils, 3 to 5 sub-samples were collected within a circle of 2 m diameter in all treatments and then were mixed to obtain a bulk sample for the plot. After air drying at the room temperature, the soil samples were ground and sieved to remove particles >2 mm, and then stored in plastic bags. The soil samples were analyzed at Swedish University of Agricultural Sciences.

2.4. Soil analysis

Total N (N_{tot}) and total organic carbon (TOC) were determined on finely ground samples on a LECO CHN analyzer (Leco CHN®CHN 932 analyzer). Prior to the analyses, the samples were treated by 4M HCl (1:1 soil:solution ratio) for dissolution of carbonates. The soil EC and pH were measured in deionized H_2O (1:5 soil:solution ratio), and pH_{CaCl2} was determined after adding 0.5M $CaCl_2$ [27]. The soil samples were extracted with 1M NH_4NO_3 for 2 hours (1:2.5 soil:solution ratio) to quantify the exchangeable and specifically adsorbed fraction of trace metals (i.e. Cd, Cu, Pb, Zn) [2, 22]. Potentially dissolved metals were extracted with 0.025 M Na_2H_2EDTA (1:10 soil:solution ratio) for 1.5 hours [28]. The reverse *aqua regia* (3:1 HNO_3:HCl ratio)-digestible fraction (Rev *Aq Reg*) of Cd, Cu, Pb and Zn was extracted by using a method described by Stevens et al. [27]. After centrifugation, filtration and dilution (if necessary), the metal concentrations were determined by using inductively coupled plasma-mass spectrometry (ICP-MS, Perkin Elmer ELAN 6100).

2.5. Biosolids sampling and analyses

Biosolids samples (one sample of chicken manure, and one sample of composted manure) were sampled at the time of application and analyzed for dry matter content, total organic carbon, N, P, K and trace metals (Cd, Cu, Pb, Zn) by NISF (Table 2) [8]. Total organic carbon (TOC) was determined by the Walkley-Black method [30], total N (N_{tot}) was determined by using the Kjeldahl procedure [18], while concentrated HNO_3 and H_2SO_4 digestion [14] was used for total P and K. Digests were neutralized by adding NH_4OH (10%), P was determined colorimetrically [3] and K - by flame emission spectrometry. Trace metal concentrations (Cd, Cu, Pb, Zn) of biosolids were determined after digestion using a Rev *Aq Reg* procedure [27]. Copper, Pb and Zn were determined on filtered digest samples using flame atomic absorption (AAS, Perkin Elmer 3300) and Cd with a graphite furnace AAS [8].

Table 2. Characteristics of the biosolids (chicken manure and composted manure) used in the experiments in Vinh Phuc and Ha Tay provinces [8]

No.	Parameters	Units	Chicken manure	Composted manure[a]
1	Moisture	%	57	53
2	Total nitrogen (N_{tot})	%, dw	1.6	1.3
3	Total phosphorus (P_{tot})	%, dw	1.1	1.2
4	Potassium (K)	%, dw	1.6	2.3
5	Total organic carbon (TOC)	%, dw	31.4	18.2
6	Copper (Cu)	mg kg^{-1}, dw	48.5	45.0
7	Zinc (Zn)	mg kg^{-1}, dw	263.0	190.0
8	Cadmium (Cd)	mg kg^{-1}, dw	3.4	3.5
9	Lead (Pb)	mg kg^{-1}, dw	25.3	15.9

[a] Composted manure = composted mixture of pig manure and rice straw. dw = dry weight.

2.6. Statistical analysis

Data from the experiments were analyzed using the General Linear Model (GLM) procedure of Minitab Software version 14.0 [19]. Treatment means which showed significant differences at the probability level of P<0.05 were compared using Tukey´s pairwise comparison procedure, while the biosolids application was used as factor in the models. The statistical model used was $y_{ij} = \mu + \alpha_i + e_{ij}$, where μ is the mean value for all treatments, α_i is the different between mean value of treatment *i* with overall mean, and e_{ij} is a random error.

The results were also analyzed by regression analysis to assess the relationship between concentrations of elements in the soil (TOC, N_{tot}, trace metals) and the amount of biosolids applied. The statistical regression model was: $y_{ij} = a + bx_i + e_{ij}$, where y is the concentration of elements, a is the intercept, b is the slope of y_i against the corresponding value of y_i, x_i the biosolids dose, and e_{ij} is the random error effect.

3. Results

3.1. The effects of applying biosolids on soil pH and electrical conductivity

The application of biosolids showed a tendency to increase of soil pH, an effect that was significant at the higher application rates (Table 3). Significant effects on EC were found for the high biosolid application rates of 63 t ha^{-1}

(tons per hectare) and 60 t ha^{-1} for composted manure and chicken manure respectively (450% and 300% of normal application rate respectively). The reason of increasing soil pH and EC might is due to increased soil organic matter (see below) and alkali-metals at higher application rates of biosolids.

Table 3. Electric conductivity (EC, $\mu S\ cm^{-1}$), pH, exchangeable Ca, Mg, Na, K (1M NH_4NO_3 extractable; g kg^{-1}) in topsoil (0-20 cm) samples from experiments. Different superscript letters indicate significant differences between treatments at the same site (P<0.05)

Site	EC[1]	$pH^1_{H_2O}$	$pH^2_{CaCl_2}$	Exchangeable			
				K	Na	Ca	Mg
Ha Tay site							
BoDp1	62.78[a]	6.02[a]	5.23	0.07	0.02	1.36	0.17
BoDp2	61.85[a]	6.05[a]	5.35	0.07	0.02	1.36	0.18
BoDp3	63.69[a]	5.99[a]	5.23	0.07	0.02	1.33	0.17
BoDp4	67.32[a]	6.08[b]	5.31	0.08	0.02	1.39	0.18
BoDp5	66.20[a]	6.13[b]	5.37	0.06	0.02	1.42	0.18
BoDp6	71.39[b]	6.13[b]	5.35	0.06	0.02	1.43	0.19
Vinh Phuc site							
BoMl1	39.32[a]	6.08[a]	5.37[a]	0.02[a]	0.01[a]	0.52[a]	0.03[a]
BoMl2	49.25[a]	6.36[a]	6.43[a]	0.02[a]	0.01[a]	0.71[a]	0.06[a]
BoMl3	38.61[a]	6.25[a]	5.54[a]	0.02[a]	0.01[a]	0.55[a]	0.04[a]
BoMl4	41.39[a]	6.17[a]	5.35[a]	0.02[a]	0.01[a]	0.55[a]	0.05[a]
BoMl5	80.12[b]	7.65[b]	7.13[b]	0.05[b]	0.03[b]	0.86[b]	0.10[b]
BoMl6	98.25[b]	7.96[b]	7.32[b]	0.09[b]	0.05[b]	0.93[b]	0.20[b]

[1] pH in H_2O, ratio soil : water = 1:5
[2] pH in 0.05 M $CaCl_2$, ratio soil : solution = 1:5

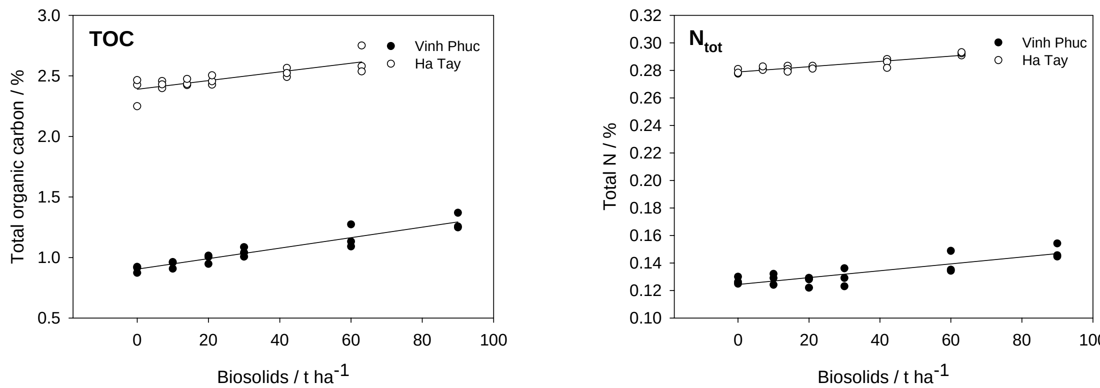


Fig. 1. Correlation between total organic carbon (TOC), total nitrogen (N_{tot}) in soil and biosolids application rate in the Ha Tay and Vinh Phuc experiments. The regression equations for Ha Tay were TOC (%) = $2.39 + 3.38 \times 10^{-3}$ t ha^{-1} composted manure ($r^2 = 0.63$) and N_{tot} (%) = $0.28 + 0.19 \times 10^{-3}$ t ha^{-1} composted manure ($r^2 = 0.84$). The regression equations for Vinh Phuc were TOC (%) = $0.90 + 4.33 \times 10^{-3}$ t ha^{-1} chicken manure ($r^2 = 0.89$) and N_{tot} (%) = $0.12 + 0.25 \times 10^{-3}$ t ha^{-1} chicken manure ($r^2 = 0.72$).

3.2. The effects of applying biosolids on soil organic carbon and total nitrogen contents

The application of biosolids for agriculture caused a significant increase of TOC and N_{tot} contents at all experimental sites (Fig. 1). The increase was linearly related to the amount of biosolids applied. The linear regression indicated that the slope of the relationship was higher for the Vinh Phuc site in comparison to the Ha Tay site. This was probably due to the concentration of both TOC and N_{tot} being much higher in the chicken manure than in the composted manure (Table 2).

3.3. The effects of applying biosolids on trace metal concentrations in soil

The concentrations of Rev *Aq Reg*-digestible Zn in soils were increased significantly by application of biosolids at both experimental sites. Copper concentrations were increased significantly by application of biosolids only at the Vinh Phuc site. There was a higher increase in Zn concentration at Vinh Phuc compared with Ha Tay. This was probably due to the concentration of Zn being higher in the chicken manure than in the composted manure. There were no significant effects of biosolids application on concentration of Rev *Aq Reg* Cd and Pb in treated soils (Fig. 2).

The potentially dissolved Cd, Cu, Pb and Zn (EDTA-extractable) increased linearly as a function of biosolids application, except for Pb in the Ha Tay experiment (Table 4 and Fig. 3). The NH_4NO_3-extracted fractions of Cd, Cu, Pb and Zn constituted only a small proportion of the EDTA-extracted fractions. There were no significant differences between different biosolids and application rates. The reason for this lack of significance might be the low concentrations in combination with a variation between the replicates.

Table 4. Effect of biosolids application on 0.025 M EDTA (mg kg^{-1} dw) and 1M NH_4NO_3 extractable (mg kg^{-1} dw) trace metals. Different letters indicate significant differences between treatments at the same site (P<0.05)

Site	EDTA-extractable				NH_4NO_3-extractable			
	Cu	Zn	Cd	Pb	Cu	Zn	Cd	Pb
Ha Tay site								
BoDp1	17.19^{a}	4.26^{a}	0.189^{a}	23.28	0.013	0.494	0.0287	0.075
BoDp2	17.48^{ab}	4.13^{a}	0.194^{a}	23.00	0.011	0.374	0.0241	0.055
BoDp3	17.72^{ab}	4.62^{a}	0.206^{a}	23.55	0.017	0.552	0.0328	0.077
BoDp4	17.75^{ab}	5.08^{ab}	0.209^{ab}	23.45	0.014	0.524	0.0258	0.064
BoDp5	18.44^{b}	5.04^{ab}	0.215^{ab}	24.07	0.013	0.457	0.0246	0.050
BoDp6	19.71^{c}	6.16^{b}	0.222^{b}	23.51	0.011	0.531	0.0211	0.041
Vinh Phuc site								
BoMl1	3.89^{a}	9.51^{a}	0.037^{a}	3.56^{a}	0.031	1.884	0.0048	0.012
BoMl2	4.50^{ab}	13.27^{ab}	0.046^{a}	4.05^{a}	0.057	0.216	0.0010	0.001
BoMl3	4.05^{ab}	11.02^{ab}	0.038^{a}	3.83^{ab}	0.037	1.407	0.0029	0.006
BoMl4	4.04^{ab}	11.93^{ab}	0.039^{a}	3.83^{ab}	0.039	2.088	0.0032	0.007
BoMl5	5.20^{bc}	24.09^{bc}	0.049^{ab}	4.50^{bc}	0.118	0.059	0.0004	<0.001
BoMl6	6.10^{c}	35.28^{c}	0.066^{b}	4.86^{c}	0.153	0.072	0.0003	<0.001

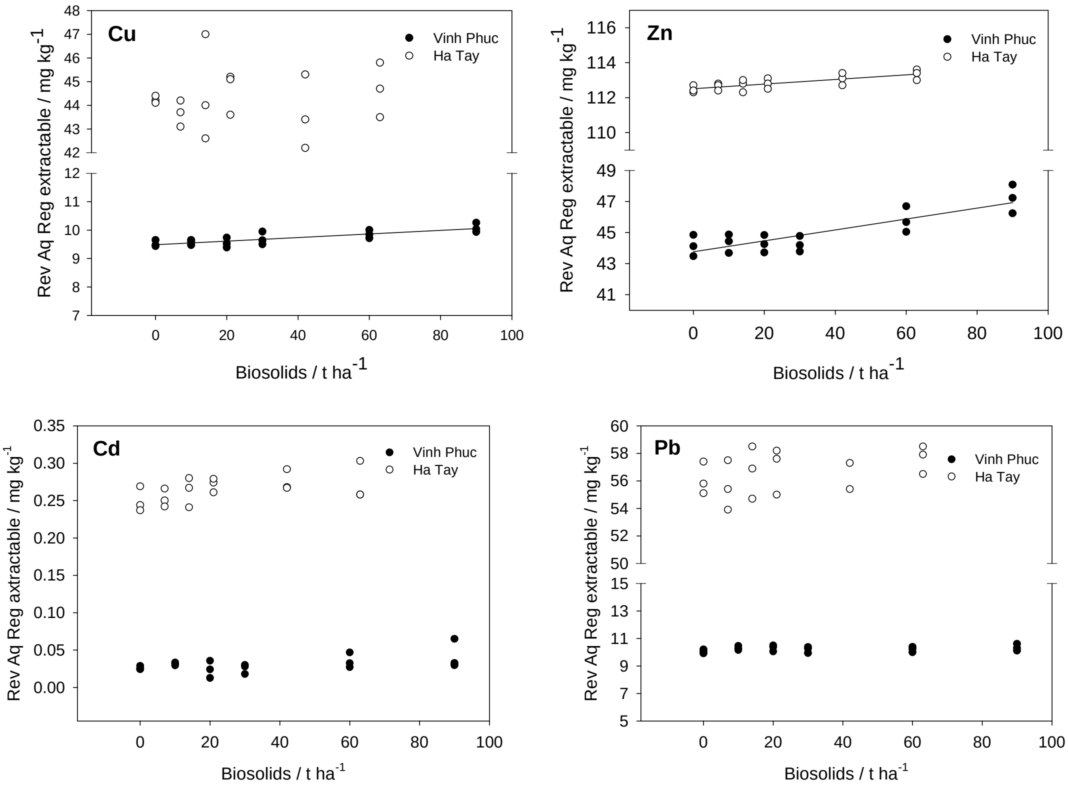
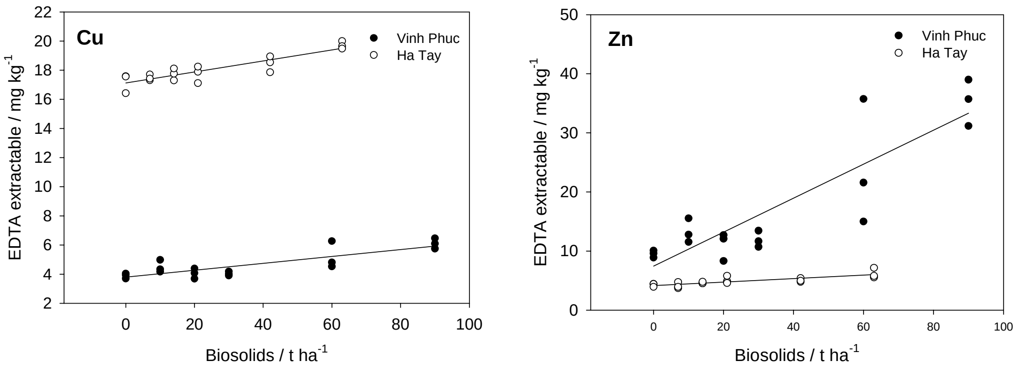


Fig. 2. Correlation between Cd, Cu, Pb and Zn (reverse *aqua regia*-extractable) in soil and biosolids application rate in the Ha Tay and Vinh Phuc experiments. Only significant results are shown with regression line. The regression equation for Ha Tay was Zn (mg kg^{-1}) = 112.51 + 13.25 $\times$ 10^{-3} t ha^{-1} composted manure (r^2 = 0.58). The regression equations for Vinh Phuc were Cu (mg kg^{-1}) = 9.48 + 6.32 $\times$ 10^{-3} t ha^{-1} chicken manure (r^2 = 0.68) and Zn (mg kg^{-1}) = 43.77 + 35.04 $\times$ 10^{-3} t ha^{-1} chicken manure (r^2 = 0.73).



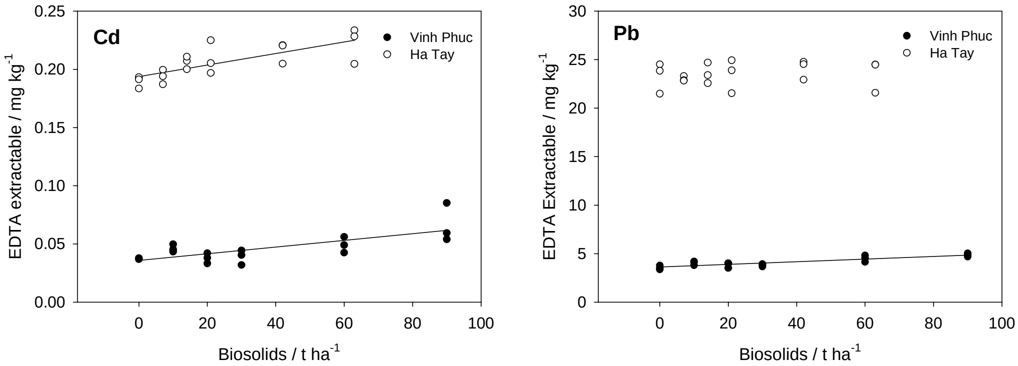


Fig. 3. Correlation between Cd, Cu, Pb and Zn (EDTA-extractable) in soil and biosolids application rate in the Ha Tay and Vinh Phuc experiments. Only significant results are shown with regression line. The regression equations for Ha Tay were Cu = 17.2 + 0.037 t ha^{-1} composted manure (R^2 = 0.78); Zn = 4.16 + 0.029 t ha^{-1} composted manure (R^2 = 0.61); Cd = 0.19 + 0.0005 t ha^{-1} composted manure (R^2 = 0.55). The regression equations for Vinh Phuc were Cu = 3.80 + 0.024 t ha^{-1} chicken manure (R^2 = 0.67); Zn = 7.45 + 0.286 t ha^{-1} chicken manure (R^2 = 0.77); Cd = 0.035 + 0.0003 t ha^{-1} chicken manure (R^2 = 0.51); Pb = 3.63 + 0.014 t ha^{-1} chicken manure (R^2 = 0.73).

4. Discussion

The use of biosolids as a fertilizer significantly increased TOC and N_{tot} in the soils. The organic matter in biosolids increases the ability of soil to retain water. The biosolids treatment involved application of 7-63 t ha^{-1} of composted manure or 10-90 t ha^{-1} of chicken manure incorporated into the top 20 cm of the soil. The topsoil bulk density was 1.2 t m^{-3} for the Ha Tay site and 1.3 t m^{-3} for the Vinh Phuc site. The theoretical estimations showed that soil organic carbon content (TOC, %) would have initially increased by a factor of 3.3×10^{-3} and 5.5×10^{-3} t^{-1} biosolids for Ha Tay and Vinh Phuc sites, respectively, as a result of the biosolids application. The field experimental data showed that the organic carbon increase for the composted manure was in agreement with the calculated value. However, for the chicken manure, the increase in measured carbon content was lower than estimated (Fig. 2). This was probably due to the decomposition of chicken manure that occurred during the experimental period, and that was less pronounced in the composted manure, which had probably already decomposed during the composting process. An increase in total organic carbon and nitrogen through application of biosolids has also been found in the previous studies [12, 31, 32]. However, Garrido et al. (2005) did not find a significant increase in organic matter and total nitrogen, possibly because a lower biosolids rate (4.5 t ha^{-1}) was used in that study [27].

Although biosolids have been demonstrated to be an useful nutrient source for agricultural soils, the beneficial properties of biosolids can, depending on their origin, be limited by their contents of potentially harmful substances. The biosolids applied in the experiments in this study did not originate from domestic waste but from animal manure. The same practice has been observed in the studies of nutrient fluxes in peri-urban vegetable production in the Asia, where the animal manure is purchased from villages specializing in animal production and transported to peri-urban areas, where it is applied in intensive agricultural production systems, in particular to vegetable crops [11, 13]. The soil amended with biosolids in the present study had higher "total" (Rev *Aq Reg*) concentrations of Cu (only for the Vinh Phuc site) and Zn than the control soils. Ogiyama et al. (2006) reported accumulation of Zn in soils due to animal manure application [20]. Sloan et al. (1997) reported that biosolids application significantly increased total concentrations of Cu and Zn in the soils studied, although the differences were less than the initial increase anticipated for the application of biosolids [25]. This may be due to plant trace metal uptake. Furthermore, the increase in trace metal contents in soil may be dependent on the biosolids application rate [6]. In the present study, there was a significant ($P<0.05$) increase in "total" Zn for the application rates greater than 21 t ha^{-1} for the composted manure and 30 t ha^{-1} for the chicken manure.

Although the "total" concentrations of Cd and Pb in the chicken manure and Cd in the composted manure were higher than in the experimental soils, the concentrations of Cd and Pb in biosolids-amended soils were not significantly higher, although there was an increasing trend. This was probably due to the concentrations of Cd and Pb in biosolids were not being sufficiently high to give significant effects on concentrations of these metals (Rev *Aq Reg*) in experimental soils. The content of Pb in composted manure was lower than that in experimental soil (Ha Tay site). In addition, the short-term nature (one time) of biosolids application in these experiments may have contributed to the lack of a significant effect on these elements.

Trace metals in biosolids are generally strongly sorbed to the biosolids matrix. Thus, trace metals added to soil with biosolids are less phytoavailable than those added as simple inorganic salts [15]. There was no significant effect on the NH_4NO_3-extractable fraction of trace metals compared with the level in control soils. However, the potential dissolved Cd, Cu and Zn was significantly higher than in the control soils, but not in the case of Pb. This indicates that adding biosolids to agricultural soils can increase binding sites or even act as a sink for trace metals already present in soil, reducing metal concentration in the soil solution, despite the biosolids having higher metal concentrations than the soil itself [1, 9, 16]. Cripps et al. (1992), who found that application of biosolids at a rate of 11 t ha^{-1} increased availability of Cu in soil, but that neither Cu nor Zn was leached from surface soil [1].

5. Conclusions

Application of biosolids as fertilizer sources has become a common practice in Vietnam, especially in the peri-urban areas. The reuse of these nutrients had some beneficial effects on soil fertility, such as increased total organic carbon and nitrogen. This study found that both organic carbon content and total nitrogen were improved in soils treated with biosolids. However, these benefits were limited by the presence of some potential toxic trace metals in biosolids.

The addition of biosolids, here in the form of chicken manure and composted manure, also increased the soil concentration of EC. The total concentrations of Zn and Cu (for the Vinh Phuc site) and potential dissolved Cd, Cu, and Zn were significantly higher in soils treated with biosolids, whereas the total concentrations of Cd and Pb were not clearly different from the control. This was probably due to the short-term nature of biosolids application (one occasion) and the relatively low concentrations of these trace metals in the biosolids.

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